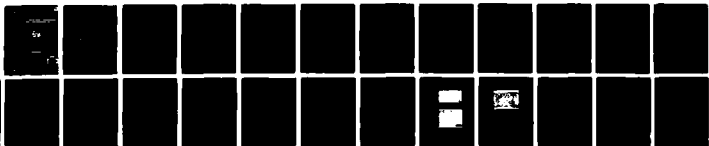


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Improvement of Transverse Strength in Composite Materials Composites by High-Strength Surface Films

G. L. STICKEL, R. G. RENDALL
and W. C. KELLY
Materials Science Laboratory
Laboratory Operations
The Aerospace Corporation
El Segundo, California

1 February 1982

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This interim report was submitted by The Aerospace Corporation, El Segundo, CA 90245, under Contract No. F04701-81-O-0082 with the Space Division, Deputy for Technology, P.O. Box 92960, Worldway Postal Center, Los Angeles, CA 90009. It was reviewed and approved for The Aerospace Corporation by S. Feuerstein, Director, Chemistry and Physics Laboratory. Major R. R. Gajewski, SD/YLXT, was the Project Officer for the Mission-Oriented Investigation and Experimentation (MOIE) Program.

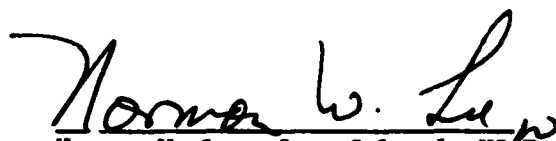
This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.


Ralph R. Gajewski, Major, USAF
Project Officer


Florian P. Meinhardt, Lt Col, USAF
Director, Directorate of Advanced
Space Development

FOR THE COMMANDER


Norman W. Lee, Jr., Colonel, USAF
Deputy for Technology

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Graphite-aluminum composites were fabricated having special high-strength surface foils. The foils employed were an aluminum powder metallurgy alloy (IN9051) and a SiC-Al composite. Both improved the transverse strength of graphite-aluminum panels above that achieved with 6061 aluminum foils. The transverse strength of panels having the IN9051 foil was three times that of panels with 6061 foils of equivalent thickness. The primary effect of the SiC-Al foil was an improved elastic modulus in the transverse and longitudinal directions. The IN9051 foils		

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had no effect on the composites' longitudinal strength, but the SiC-Al foil degraded the longitudinal strength.

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I. INTRODUCTION

The usefulness of graphite-aluminum composites is currently limited by their poor strength in the directions perpendicular to the graphite fibers. This is particularly troublesome in structures requiring a unidirectional fiber orientation. Improving the transverse strength would enhance the design versatility beyond that which is currently attainable with these materials and would make graphite-aluminum more competitive with other metal-matrix composites.

The required transverse strength is presently achieved by encapsulating the individual composite wires in aluminum foil. The transverse strength is increased by 50 to 75%, and the joinability of the composite is greatly enhanced by this procedure.¹ However, encapsulation increases production costs and diminishes the fiber content and, therefore, the longitudinal properties by 25% or more.

The ultimate solution to the transverse strength problem is to change the composite processing procedures to strengthen the fiber-matrix bond. The Aerospace Corporation is committed to this approach and has devoted considerable effort to it under the funding of the Naval Surface Weapons Center (NSWC).²⁻⁴ Although much progress has been made, the process is a formidable task and is still under development. Thus, the need for a less complicated

¹W. C. Harrigan, Jr., J. Dolowy, and B. Webb, Investigations of Joining Concepts for Graphite Fiber Reinforced Composites, Final Report, NSWC Contract N6092-77-C-0166, DWA Composite Specialties, Chatsworth, Calif. (1977).

²D. L. Dull and M. F. Amateau, Transverse Strength Properties of Graphite-Aluminum Composites, Final Report, TOR-0077(2726-03)-4, The Aerospace Corporation, El Segundo, Calif. (30 September 1977).

³G. L. Steckel, R. H. Flowers, and M. F. Amateau, Transverse Strength Properties of Graphite-Aluminum Composites, Final Report, TOR-0078(3726-03)-4, The Aerospace Corporation, El Segundo, Calif. (30 September 1978).

⁴G. L. Steckel, H. A. Katzman, and M. F. Amateau, Transverse Strength Properties of Graphite-Aluminum Composites, Final Report, TOR-0079(4726-03)-4, The Aerospace Corporation, El Segundo, Calif. (30 September 1979).

nearer term solution to the problem has become apparent. In response, a study has been conducted to determine the suitability of high-strength aluminum alloys or composites for use as cladding materials on graphite-aluminum plates. The composite consolidation procedures presently employed require aluminum surface foils. Thus, this approach would be compatible with the existing process. It would be more cost effective than encapsulation and would eliminate the associated fiber dilution. However, this approach would be limited to those applications requiring transverse strength within the plane of a composite plate. It would have no effect on the strength through the thickness of the plate.

II. EXPERIMENTAL PROCEDURE

A. SURFACE FOIL MATERIALS

International Nickel Company's IN9051 alloy and a SiC-6061 composite produced by NSWC were selected for use as high-strength surface foils for graphite-aluminum composites. IN9051 is a powder metallurgy aluminum alloy containing 4 wt. % MgO. It has a typical ultimate tensile strength of 80×10^3 psi with a yield strength of 75×10^3 psi and an elastic modulus of 11×10^6 psi. The strengthening is believed to be a result of the dispersed MgO particles. IN9051 alloy sheet stock, 0.07-in. thick, was provided by Lockheed Aircraft Company, Marietta, Georgia.

The SiC-6061 was received from NSWC in the as-extruded bar form with a 0.6-in. \times 1.5-in. cross section. It had 15 vol. % of Silag Incorporated's grade M-8 SiC, which is 15 to 25% whiskers and 75 to 85% particulate. Although the tensile properties of the starting material were not obtained, typical properties reported by Silag for their 17 vol. % M-8 SiC-6061-T6 composite are an ultimate tensile strength of 65 to 70×10^3 psi, a yield strength of 55 to 60×10^3 psi, and an elastic modulus of 14×10^6 psi. Thus, the reinforcement increases the tensile properties of the 6061-T6 alloy by 40 to 60%.

The IN9051 and SiC-6061 had to be rolled to a maximum foil thickness of 0.005 in. before fabrication of the graphite-aluminum plates. These materials have very limited ductilities (less than 6% elongation). Therefore, fairly elaborate rolling schedules were adopted for the foil preparation. Both materials were hot-rolled to 0.009 in. using a Loma mill which had flame heated, 6-in. diameter rolls. The SiC-6061 was given 10% reduction per pass and was annealed at 500°C for 10 minutes between passes. The IN9051 was somewhat more ductile than the SiC-6061 and could be given from 15 to 20% reduction per pass with intermediate anneals at 400°C for 10 minutes. Both materials were further reduced by cold-rolling, using the same reduction per pass and annealing conditions that were used for hot-rolling. The SiC-6061 was cold-rolled to a final thickness of 0.0045 in., and the IN9051 was cold-rolled to 0.0035 or 0.005 in. All foils were annealed at 400°C for 30 minutes and furnace cooled following the final rolling pass.

B. COMPOSITE PROCESSING

Nine composite plates were fabricated using graphite-aluminum precursor wire purchased from Material Concepts Incorporated. The precursor wire had Union Carbide's VSB-32 or VS0054 pitch mesophase graphite fibers in a matrix of 6061 aluminum modified by additions of Ti and B in excess of the liquid solubility limits. The properties of the graphite fibers are given in Table 1.

Table 1. Properties of Graphite Fibers

<u>Fiber</u>	<u>Tensile Strength, 10³ psi</u>	<u>Young's Modulus, 10⁶ psi</u>	<u>Density, g/cm³</u>
VSB-32	300	55	2.02
VS0054	350	100	2.05

The VSB-32/6061 precursor wire had a fiber content of 50 vol. % and an average ultimate tensile strength of 150×10^3 psi, or 97% rule of mixtures (ROM). The VSB-54/6061 wire had 40 vol. % fiber with a tensile strength of 130×10^3 psi (88% ROM).

DWA Composite Specialities fabricated the unidirectional graphite-aluminum plates by the diffusion bonding process. The composite plates are provided in Table 2 along with the plate dimensions, foil thicknesses, and the number and type of mechanical property samples tested.

Table 2. Graphite-Aluminum Composite Plates

Composite Fiber/Matrix/Foil	Plate Number	Length-Width-Thickness, In.		Foil Thickness, In.	Tensile Samples ^a
VS8-32/6061(1L)/6061	G4210	12 x 12 x 0.026		0.0035	12LT, 4LTR, 4TT
VS8-32/6061(1L)/SiC-6061	G4261	10 x 6 x 0.030		0.0045	4LT, 3LTR, 4TT
VS8-32/6061(1L)/IN9051	G4272	10 x 6 x 0.030		0.005	4LT, 3LTR, 4TT
VS8-32/6061(2L)/IN9051	G4407	3.6 x 1.8 x 0.047		0.005	2LT, 5TT
VS8-32/6061(3L)/IN9051	G4404	4 x 2 x 0.065		0.005	2LT, 3TT
VS0054/6061(1L)/6061	G4323	12 x 12 x 0.029		0.0035	4LT, 4LTR, 4TT
VS0054/6061(1L)/IN9051	G4452	5.9 x 1.6 x 0.026		0.0035	4LT, 2LTR, 5TT
VS0054/6061(1L)/IN9051	G4405	3.7 x 1.7 x 0.029		0.005	2LT, 5TT
VS0054/6061(2L)/IN9051	G4406	3.2 x 1.6 x 0.048		0.005	1LT, 5TT

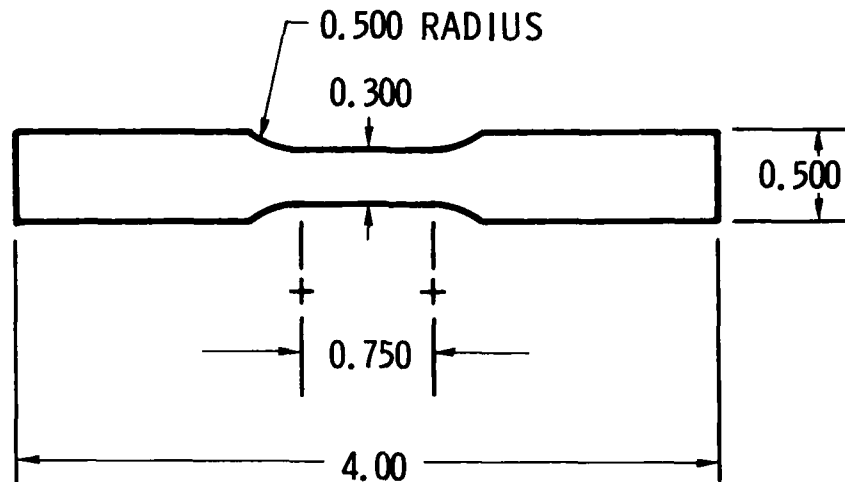
^aLT = Longitudinal Tensile Sample (no reduced section)

LTR = Longitudinal Tensile Sample with Reduced Section

TT = Transverse Tensile Sample

C. MECHANICAL PROPERTIES EVALUATION

Ultimate tensile strength and Young's Modulus were determined for the IN9051 and SiC-6061 foils and for the composite plates. The foils were tested using the sample geometry shown in Figure 1. Two types of sample geometry



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Figure 1. Foil and Composite Longitudinal Tensile Sample Geometry.

were used for the longitudinal composite tensile samples. All the plates were tested using rectangular samples, which were the full-plate thickness and approximately 4-in. long by 0.25-in. wide. The larger plates (see Table 2) were also tested using the same sample geometry that was used for the foils. It was learned that the measured longitudinal tensile strength of the composites with the VS0054 fiber was a function of the sample geometry.

The transverse tensile samples were 4-in. long by 1-in. wide for the larger plates (G4210, G4261, G4272 and G4323) and 1.5-in. by 0.5-in. for the remainder of the plates. Young's modulus was determined for the larger transverse samples and all of the longitudinal samples using a clip-on extensometer. The modulus was not measured for the small transverse samples. Aluminum

tabs 0.05-in. thick were bonded to both sides of each end of the composite tensile samples to reduce stress concentration effects of the Instron testing machine grips.

The fiber content and aluminum carbide (Al_4C_3) concentrations of the composite plates were determined by gas chromatography. The tensile testing and gas chromatography were performed following the procedures outlined by Padilla et al.⁵

⁵F. Padilla, W. C. Harrigan, Jr., and M. F. Amateau, Handbook of Test Methods for Evaluation and Qualification of Aluminum-Graphite Composite Materials, Interim Report, TOR-0075(5621)-3, The Aerospace Corporation, El Segundo, Calif. (21 February 1975).

III. RESULTS AND DISCUSSION

A. FOIL MECHANICAL PROPERTIES

The tensile data for the cold-rolled and annealed SiC-6061 and IN9051 foils are provided in Table 3 along with data for the annealed 6061 alloy. Although the tensile strength of the as-received IN9051 was not determined, the typical tensile strength of the material is 80×10^3 psi. Thus, the strength of the IN9051 may have been reduced by up to 10% by the rolling and annealing. The material was given a second anneal at 600°C to simulate the diffusion bonding process. This anneal reduced the tensile strength by 10% to 64.5×10^3 psi. The tensile strength of the IN9051 did not depend on the foil orientation with respect to the rolling direction.

Table 3. Mechanical Properties of SiC-6061 and IN9051 Foils

<u>Foil</u>	<u>Tensile Strength,</u> <u>10^3 psi</u>	<u>Young's Modulus</u> <u>10^6 psi</u>
6061-0	18.1	10.0
SiC-6061-0	36.1	11.0
IN9051(400°C anneal)	71.5	10.0
IN9051(600°C anneal)	64.5	--

No data were available for the as-received SiC-6061 material in the annealed condition. Therefore, it was not possible to determine if the rolling had any effect on the material's tensile properties. However, since the tensile strength of the annealed foil was twice that of 6061-0, compared to a typical increase of 40 to 50% for extruded SiC-6061-T6, it is unlikely that the rolling had a deleterious effect on the strength of the SiC-6061. The SiC-6061 was only tested in the rolling direction.

The moduli of the foils were somewhat lower than anticipated. However, the accuracy of these values is questionable because it is very difficult to make accurate strain measurements with extensometers on thin foils.

B. TRANSVERSE STRENGTH OF GRAPHITE-ALUMINUM COMPOSITES

The transverse strength data for the graphite-aluminum composites are given in Table 4 and are plotted as functions of the fractional foil

Table 4. Transverse Strength of Graphite-Aluminum Composites with Various Surface Foils

Composite Fiber/Matrix/Foil	Fractional Foil Thickness, In.	Transverse Strength,	
		10^3 psi	% ROM
VSF-32/6061(1L)/6061	0.26	4.8	78
VSF-32/6061(1L)/S1C-6061	0.30	7.9	65
VSF-32/6061(1L)/IN9051	0.33	18.2	80
VSF-32/6061(2L)/IN9051	0.21	10.0	71
VSF-32/6061(3L)/IN9051	0.15	8.1	86
VS0054/6061(1L)/6061	0.26	5.3	86
VS0054/6061(1L)/IN9051	0.27	13.1	69
VS0054/6061(1L)/IN9051	0.35	17.7	74
VS0054/6061(2L)/IN9051	0.21	10.1	67
VSF-32/6061(3L)/6061	0.18	3.3	68

thickness, t_f , in Figure 2. The fractional foil thickness was defined as the ratio of the foil thickness to the total plate thickness. The ROM data in the table were calculated from the equation

$$\% \text{ ROM} = 100\sigma / [t_f \sigma_f + (1-t_f) \sigma_c]$$

where σ is the measured plate transverse strength, σ_f is the foil strength given in Table 3, and σ_c is the transverse strength of the composite without surface foils. A value of 2×10^3 psi was assumed to be a reasonable value

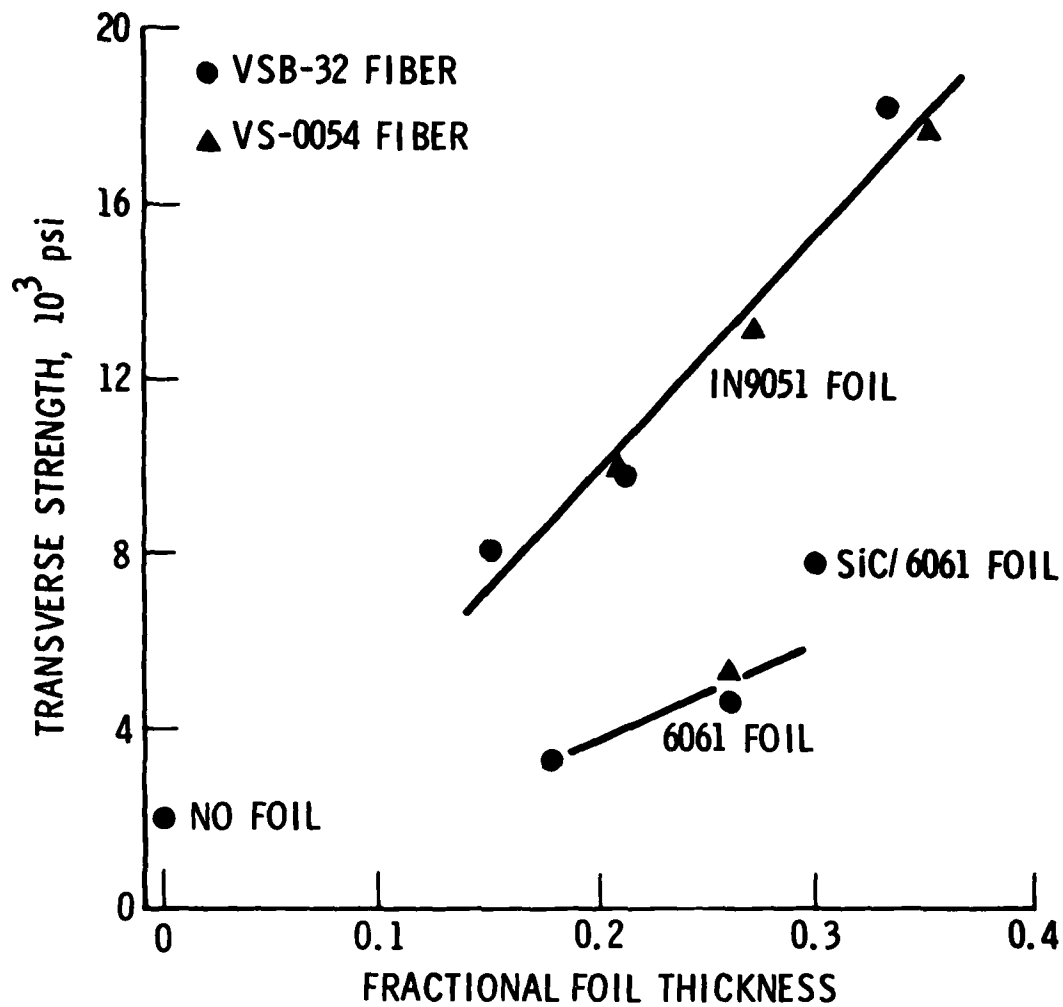


Figure 2. Transverse Strength of Graphite-Aluminum as a Function of the Surface Foil Type and Thickness

for σ_c for both composite systems.³ The rule of mixtures strength of the composite with the SiC-6061 foil was 65% ROM, while the composites with the 6061 and IN9051 foils varied from 68 to 86% ROM and 66 to 80% ROM, respectively. Thus, relative to the foil tensile strength, the three foils were similarly effective in increasing the composite transverse strength. The fairly consistent ROM values and the linearity of the plots in Figure 2 allow the transverse strength to be estimated with reasonable accuracy from the fractional foil thickness and foil tensile strength.

The plots in Figure 2 demonstrate the superior transverse strength of the composites with the IN9051 foil. The figure also indicates that high-strength foils, such as IN9051, will provide transverse strength while maintaining a minimal foil fraction. Thus, it will not be necessary to decrease the fiber content and, therefore, the longitudinal properties in order to meet the transverse strength requirements. Furthermore, for some applications, high-strength foils should permit the use of thinner composite panels, thus enhancing the weight-saving potential of the composite.

In many applications, the transverse yield strength may be more important than the ultimate tensile strength. Any yielding in the transverse direction will, at the very least, cause microcracking, which can have a deleterious effect on important longitudinal properties such as the coefficient of thermal expansion. Stress-strain curves are shown in Figure 3 for VSB-32/6061(IL) composites having each of the three foils. Yielding of the composite having the 6061 foil began at 3.5×10^3 psi, but did not occur until stresses of 6.5×10^3 psi and 8.5×10^3 psi were reached in the composites with the SiC-6061 and IN9051 foils, respectively. Furthermore, significant yielding did not occur with the IN9051 foil below approximately 12×10^3 psi. The effect is somewhat overemphasized in the figure, since the fractional foil thicknesses of the IN9051 and SiC-6061 are higher than that of the 6061. However, the IN9051 and SiC-6061 are high-yield strength materials, particularly when compared to the annealed 6061 alloy. Thus, it is believed that at equal fractional foil thicknesses the yield strength improvement of these foils over 6061 should be at least as great as the ultimate tensile strength improvement.

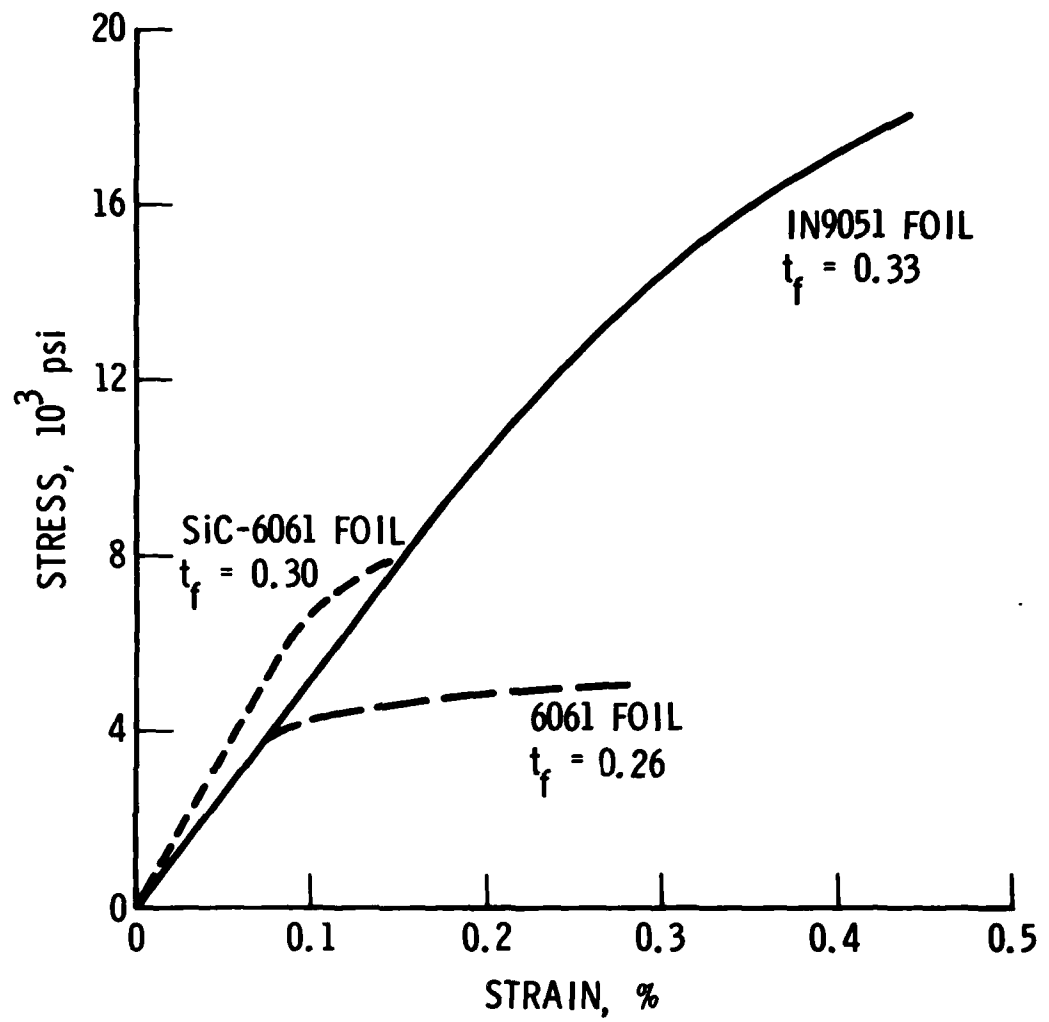


Figure 3. Transverse Stress-Strain Curves for Graphite-Aluminum Composites with Various Surface Foils

A final point apparent from Figure 3 is the transverse modulus improvement provided by the SiC-6061 foil. This foil increased the modulus from approximately 4.5×10^6 psi to 6.5×10^6 psi. The IN9051 foil had no effect on the transverse modulus.

C. LONGITUDINAL PROPERTIES OF GRAPHITE-ALUMINUM COMPOSITES

The longitudinal tensile data for the graphite-aluminum composites are provided in Table 5. The ROM calculations were made on the basis of foil contributions to the tensile properties as well as the usual fiber and matrix contributions. All but one of the composites with the IN9051 foil had lower ROM tensile strengths than the standard composites with the 6061 foil. However, it is believed that the tensile strength data for two of these composites are erroneous. None of the tensile samples of plates G4404 through G4407 had a reduced section, and it was learned that this can have a significant influence on the magnitude of the measured tensile strength. For example, the strength of plate G4323 was 109×10^3 psi (87.5% ROM) for samples having a reduced section, but only 88.8×10^3 psi (71.3% ROM) for samples which did not have a reduced section. This reduction of nearly 20% was probably the result of stress concentration effects from the testing machine grips. The dependence of the measured tensile strength on the sample geometry was even greater for plate G4452. Both of these plates had the VS0054 fiber. The tensile strength of plates G4210, G4261, and G4272, all of which had the VSB-32 fiber, did not depend on the sample geometry. Thus, the stiffer but more fragile VS0054 fiber is apparently more sensitive to stress concentration effects than is the VSB-32 fiber. It is therefore believed that the low tensile strength values reported for plates G4405 and G4406 are probably erroneous, while those for plates G4404 and G4407 are probably valid. The reason for the low strength of these plates, particularly G4407, is not known.

Pitch fiber graphite-aluminum composites usually have Young's moduli values in the 95 to 100% ROM range. Therefore, all of the composites with the IN9051 foil, except G4452, had low moduli. These plates also had poor bonding between the composite wires and the IN9051 foils. Figure 4a shows a transverse micrograph of plate G4272, which had the poorest foil bonding. Flow of

Table 5. Longitudinal Tensile Properties of Graphite-Aluminum Composites with Various Surface Foils

Composite Fiber/Matrix/Foil	Plate Number	Component Contents Fiber/Matrix/Foil, Vol%	Tensile Strength 10 ³ psi	% ROM	Young's Modulus 10 ⁶ psi	% ROM
VSF-32/6061(1L)/6061	G4210	36.3/63.7	85.8	71.3	25.4	96.5
VSF-32/6061(1L)S1C-6061	G4261	35.0/35.0/30.0	64.0	52.4	27.4	>100
VSF-32/6061(1L)IN9051	G4272	34.3/32.4/33.3	94.2	72.3	22.2	86.5
VSF-32/6061(2L)/IN9051	G4407	38.9/39.8/21.3	94.3	68.5	22.1	79.7
VSF-32/6061(3L)/IN9051	G4404	41.6/43.0/15.4	74.4	52.2	22.8	79.0
VSO054/6061(1L)/6061	G4323	32.1/67.9	109	87.5	46.8	>100
VSO054/6061(1L)/IN9051	G4452	29.7/43.4/26.9	101	78.1	40.6	>100
VSO054/6061(1L)/IN9051	G4405	28.8/36.7/34.5	83.4	64.2	33.5	92.4
VSO054/6061(2L)/IN9051	G4406	33.8/45.4/20.8	88.7	63.4	37.4	92.1

the foil was insufficient to fill in the triangular voids at the intersection between two wires and the foil. Poor bonding between the foil and composite inhibits stress transfer, and thereby decreases the measured elastic modulus. DWA Composite Specialties fabricated plate G4452 at a temperature approximately 20°C higher than that of the other plates with the IN9051 foil. It had excellent bonding between the foil and wires (Figure 4b) and a modulus that was 100% ROM. The lower fabrication temperature used for most of the plates with the IN9051 foil was the temperature normally used for plates with the 6061 foil. Thus, a slightly higher consolidation temperature is necessary with the IN9051 alloy than is with the 6061 alloy, in order to allow sufficient foil flow to achieve good bonding.

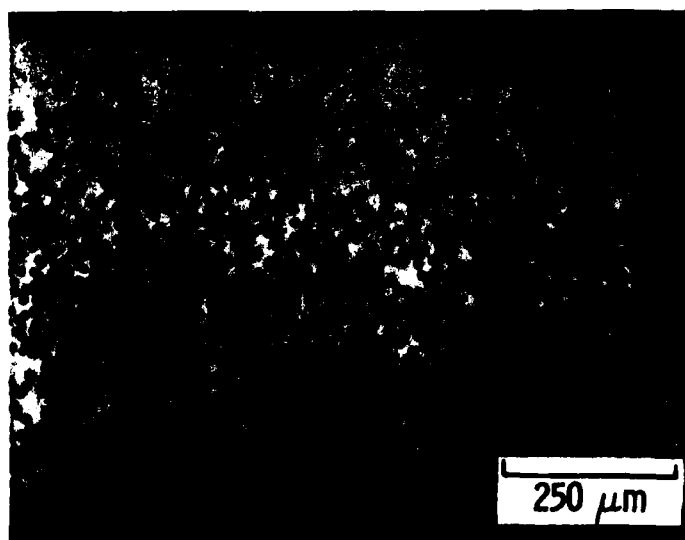
The plate with the SiC-6061 foil had the lowest absolute tensile strength and the second lowest ROM strength. The strength did not depend on the sample geometry, and bonding between the foil and composite wires appeared to be excellent. Furthermore, the modulus exceeded 100% ROM. Thus, the graphite fibers were not damaged during the consolidation process. The SiC whiskers or particles may have had a damaging effect on the graphite fibers during the tensile test; more plates must be fabricated with this foil to determine if the low tensile strength is a typical property.

Despite a lower graphite fiber content, the composite with the SiC-6061 foil had a modulus nearly 10% higher than the VSB-32/6061 standard composite. Thus, the SiC-6061 foil increased the elastic modulus in the transverse and longitudinal directions of the composite. However, the foils were very narrow strips and had to be oriented on the plate so that the rolling direction of the foil coincided with the direction of the applied load for both sample orientations. Since it was only possible to test the foils in the rolling direction, the relevance of the foil orientation to the composite properties is unknown.

Optical microscopy of plate G461 (Figure 5) indicated that there was excessive flow of the SiC-6061 foil and very little deformation of the precursor wires during consolidation. This behavior reduced the effective foil thickness and probably limited the extent of the transverse tensile



a. PLATE G4272



b. PLATE G4452

Figure 4. Transverse Optical Micrographs of Graphite-Aluminum Composites with the IN9051 Foil



Figure 5. Transverse Optical Micrograph of the Graphite-Aluminum Composite with the SiC-6061 Foil

strength and modulus improvements provided by the foil. Further tests on composites with the SiC-6061 foil must be performed to address the foil orientation issue, to determine the effect of varying the consolidation parameters, and to determine if the longitudinal strength can be improved.

All of the composites with the IN9051 foil had very high Al_4C_3 concentrations, in the range from 2000 to 4500 wt ppm. This was surprising since the foil was not expected to affect fiber-matrix reactions. We therefore did an analysis to determine the Al_4C_3 content of the IN9051 starting material. It had nearly 25,000 ppm (0.025 wt%) Al_4C_3 . It is assumed that the carbon originated from an organic binder used during the powder processing procedure. The strength of the IN9051 may be partially attributed to the high carbide concentration. The carbide may also contribute to the low ductility of IN9051.

IV. CONCLUSION

The following conclusions can be made from the study of graphite-aluminum composites with high-strength surface foils:

1. IN9051 and SiC-Al can be rolled to thin foils by applying hot and cold rolling procedures. The rolling does not adversely affect the strength of these materials.
2. The transverse strength of graphite-aluminum composites with IN9051 foils is approximately three times that of composites with 6061 foils of the same thickness.
3. SiC-6061 composite foils improve the transverse strength of graphite-aluminum, but to a lesser extent than IN9051 foils. Further study of the use of SiC-6061 foils is needed.
4. SiC-6061 foils improve Young's modulus of graphite-aluminum composites above that attained with 6061 foils in the longitudinal and transverse directions. IN9051 foils have no effect on the modulus.

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